Analyze Trace Amounts of Solvents in Water Using Packed Column GC

Glass or stainless steel columns containing 80/120 Carbopack B/3% SP-1500 packing can be used to analyze water-based samples for ppm to low percentage levels of many organic compounds. This bulletin describes minimum detection limits and maximum capacity of these columns, adsorption and memory effects, and other factors that affect component quantitation. Problems are defined that might arise during trace analyses of compounds in aqueous samples, and recommendations are made for solving these problems.

Key Words:

• solvents • water • packed column GC

Chromatographers generally express concern about analyzing aqueous samples for trace levels of solvents or other organic compounds, and with good reason. The water in such samples can reduce the life of most GC columns or coelute with sample peaks and interfere with the analysis. On the other hand, glass or stainless steel columns[•] containing 80/120 Carbopack[™] B/3% SP[™]-1500, when used for these analyses, offer excellent separations, stability, and long column life. For example, 10ppm concentrations of alcohols and ketones in water can be analyzed with excellent peak shape and response (Figure A). Table 1 shows retention times for many other compounds on this packing.

Limits for Sample Components

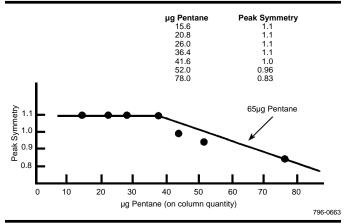
The range over which a solvent can be detected on 80/120 Carbopack B/3% SP-1500 is 0.9ng to 65µg. Using a pentane solution and a flame ionization detector (FID), we determined the sample capacity of the packing. This was done graphically by plotting the quantity of pentane injected versus peak symmetry at 10% of peak height, and noting where peak asymmetry exceeded an acceptable level (Figure B). A peak with an asymmetry value between 0.9 and 1.1 was considered symmetrical.

In determining the lower limit for detecting solvents in water by using Carbopack B/3% SP-1500 (again using an FID), we established calibration curves for glass and stainless steel columns. Peak areas were determined for various concentrations of isopropyl and sec-butyl alcohol, acetone, and methyl ethyl ketone (MEK). Peaks for each solvent were symmetrical, even at trace levels. To evaluate the linearity of peak response, we plotted the peak area versus solvent concentration data and made a least squares determination for the best line to fit the data. The correlation coefficient,

*Throughout this study, Supelco™ TightSpec glass and Supelco premium grade stainless steel were used as the column tubing materials.

Figure A. 10ppm Solvents in Water Packing: 80/120 Carbopack B/3% SP-1500 11813 (packing) 2m x 2mm ID TightSpec[™] glass Cat. No.: Column: Oven: 100°C Carrier: nitrogen, 20mL/min FID. 250°C Det .: 1µL water containing 10ppm each solvent, 200°C Inj.: Water 2 Methanol 3. Ethanol 4. Acetone Isopropyl alcohol n-Propyl alcohol Methyl ethyl ketone 6. 7. I 3 2 5 Min 796-0662







ISO 9001 REGISTERED

Table 1. Retention Times for Organic Compounds on 80/120 Carbopack B/3% SP-1500 Packing

Industrial Compounds	t _R (min)	Industrial Compounds	t _R (min)	C1-C5 Hydrocarbons	t _R (min)
Methane	1.06	DMF	21.99	Isobutylene	9.7
Ethylene	1.33	n-Valeraldehyde	22.32	Butadiene-1,3	10.6
Acetylene	1.40	Hexane	22.69	n-Butane/cis-Butene-2	10.9
Ethane	1.54	3-Pentanol	23.22	trans-Butene-2	11.6
Methanol	2.39	n-Propyl acetate	23.75	Isopentane	18.6
Propylene	2.68	Ethyl acrylate	23.92	Pentene-1	18.9
Propyne	2.78	Methyl methacrylate	24.48	n-Pentane	21.0
Acetaldehyde	2.78	Isopentanol	24.81	II I Chiane	21.0
Propane	2.91	1-Pinanol	26.42	Hazardous Solvents	t _R (min)
Methyl formate	3.80	Ethyl isopropyl ketone	26.96	Chloroform	14.6
Acetonitrile	4.55	sec-Butyl acetate	28.40	Pentane	
Ethanol	4.60	2-Methyl pentanol	28.49		15.2
Isobutane	5.70	Dimethyl acetamide	29.03	1,1,1-Trichloroethane	18.3
Propionaldehyde	6.37	Cyclohexanone	29.40	1,4-Dioxane	18.6
	6.73	2-Methyl-3-pentanol	29.66	Carbon tetrachloride	19.9
Acetone	7.54		30.00	Isopropyl acetate	21.4
Acrylonitrile		Isobutyl acetate		Benzene	21.8
n-Butane	7.06	Propyl propionate	31.33	Hexane	23.9
Methylene chloride	7.08	Ethyl methacrylate	31.39	1,1,2-Trichloroethane	24.4
Isopropyl alcohol	7.97	Isohexanol	31.71	Methyl isobutyl ketone (MIBK)	28.3
Ethyl formate	8.71	n-Butyl acetate	31.90	Methyl isoamyl ketone (MIAK)	36.4
Methyl acetate	8.71	Toluene	31.90		
Carbon disulfide	9.43	Mesityl oxide	31.90	Volatile Wastewater Pollutants	t _R (min)
n-Propyl alcohol	9.84	Heptane	31.94	Methanol/Water	2.8
Isobutyraldehyde	11.50	1-Hexanol	33.98	Methylene chloride	7.9
Ethyl ether	11.53	Cellosolve acetate	36.10	1,1-Dichloroethylene	10.5
Ethyl vinyl ether	11.53	Butyl Cellosolve	36.80	Bromochloromethane	12.2
tert-Butyl alcohol	12.02	Isoamyl acetate	37.60	trans-1,2-Dichloroethylene	13.1
THF	12.90	Ethylbenzene	38.40	1,1-Dichloroethane	13.1
Chloroform	13.34	n-Butyl acrylate	39.20	Chloroform	15.0
n-Butyraldehyde	13.38	Isoheptanol	39.60	1,2-Dichloroethane	16.0
MEK	13.50	Octane	39.98	Carbon tetrachloride	20.4
Freon [®] TF	14.11	1-Heptanol	41.44		-
Pentane	14.68	m-Xylene	41.50	Bromodichloropropane	20.4
Methyl Cellosolve®	14.69	p-Xylene	42.50	1,2-Dichloropropane	22.4
Vinyl acetate	14.94	o-Xylene	43.40	Benzene	22.4
sec-Butyl alcohol	15.09	Nonane	52.20	trans-1,3-Dichloropropene	22.4
Ethyl acetate	15.54	Decane	76.75	Chlorodibromomethane	22.4
2-Nitropropane	18.00	Decourie	10.10	cis-1,3-Dichloropropene	24.3
n-Butyl alcohol	18.36	C1-C5 Hydrocarbons	t _R (min)	Trichloroethylene	25.7
Crotonaldehyde	18.36	Methane	1.21	1-Chloro-2-bromopropane	27.5
2-Methyl pentane	19.19	Ethylene	1.61	Bromoform	31.2
Ethyl Cellosolve	19.78			1,1,2,2-Tetrachloroethylene	33.0
Benzene	20.16	Ethane	1.86	Toluene	33.0
Isovaleraldehyde	20.16	Propylene	3.7	1,4-Dichlorobutane	34.4
5	20.22	Propane	4.1	1,1,2,2-Tetrachloroethane	34.4
3-Methylpentane 3-Methyl-2-butanol	-	Isobutane	8.7	Chlorobenzene	36.2
3-10/0470/0-2-01/2001	21.88	Butene-1	9.3	Ethylbenzene	39.4

Packing: 80/120 Carbopack B/3% SP-1500*

Oven: 70° to 235°C at 4°C/min (industrial compounds, hazardous solvents, volatile pollutants) or 40° to 235°C at 4°C/min (C1-C5 hydrocarbons) and hold Carrier: nitrogen, 24cc/min (industrial compounds, C1-C5 hydrocarbons) or 20mL/min (hazardous solvents, volatile pollutants) Det.: FID

*Pre-packed columns for many instruments are available (see Ordering Information).

0.999, indicates the data points fall on a straight line. Thus, the detector response was linear for both alcohols and ketones, even at extremely low solvent concentrations. The relative standard deviation (RSD) for the area count at each concentration ranged from 1 to 4%. This low RSD at each level indicated there was no component carry-over or "memory" from one injection to another.

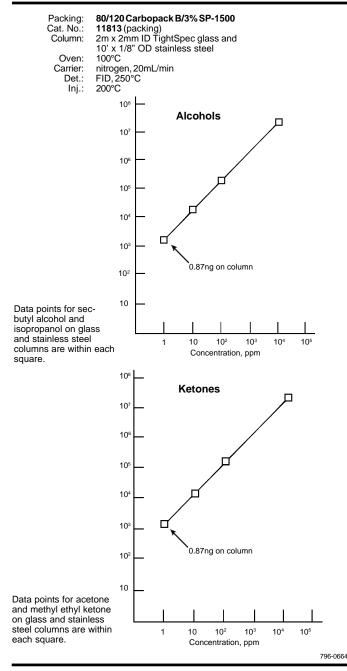
The detector response was almost identical for alcohols and ketones analyzed on glass and stainless steel columns (Figure C). This, however, does not mean that all glass and stainless steel tubing will provide equally good results. Only chromatography grade stainless steel or deactivated glass tubing should be used for analyzing trace components in aqueous samples.

The limiting factor in analyzing trace levels of alcohols and ketones in water proved to be the sensitivity limit of the FID. In these experiments we operated the chromatographic system at 2×10^{-12} AFS, close to maximum sensitivity. According to Gill and Hartmann (1), the minimum detectable peak height for an FID is 2×10^{-14} amperes, or twice the signal to baseline noise level. Using data obtained for ketones at the 1ppm level (Figure D) and extrapolating by Gill and Hartmann's procedure, we calculated that we were detecting peaks representing 2.4 x 10^{-14} amperes. Therefore, further dilution would have put the ketone concentration below the minimum detection limit of the FID. With more sensitive detection systems, samples containing less than 1ppm (0.0001% vol/vol) might be analyzed successfully.

Cat. No.: 11813 (packing)

Column: 10' x 1/8" OD stainless steel*

Figure C. Linear Response for Alcohols and Ketones on 80/120 Carbopack B/3% SP-1500

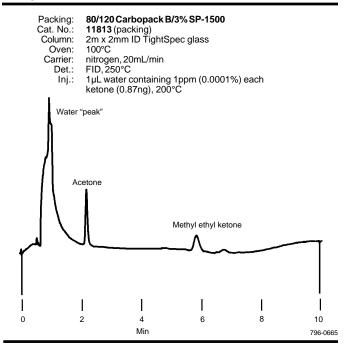


Effects of Water on Packing Stability

Repeated injections of aqueous solutions do not harm Carbopack B/3% SP-1500 packing. Column efficiency, column polarity, and peak retention times (chromatographic parameters that are sensitive to changes in column characteristics) were unchanged after rigorous column testing. To determine the effects of prolonged exposure to water on Carbopack B/3% SP-1500 packing, we injected over 200 aliquots of an aqueous solution of six solvents (approximately 10ppm each) onto a glass column and a stainless steel column, each filled with the packing. We monitored column efficiency, column polarity, and solvent retention times, and compared data from the first four injections to data from the last four injections (Table 2).

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Figure D. Trace Levels of Ketones in Water, Analyzed Without Extraction



If water were to damage the packing, **column efficiency** would decrease. No such decrease was observed. In fact, column efficiency increased slightly during the investigation (Table 2).

The relative retention times of compounds depend on the specific chemical properties of the stationary phase (column polarity). A shift in relative retention times, therefore, would indicate a chemical change in the packing. Relative retention times were monitored for two solvents, isopropyl alcohol and MEK. Since these were unchanged after 200 injections (Table 2), column polarity was unaffected by water.

If water were to strip the phase from the Carbopack column, or otherwise affect the column stability, **absolute retention times** would shift. The data in Table 2 indicate insignificant shifting of retention times (i.e., from 12.87 to 12.97 minutes, a shift of only six seconds) after 200 injections. Shifts of this magnitude often occur from day to day, or even between sequential injections.

Effects of Water on Solvent Quantification

In analyses of aqueous samples on Carbopack B/3% SP-1500, the "peak" attributed to water appears early on the chromatogram. If a thermal conductivity detector (TCD) is used, the water peak is large and will mask peaks for some low molecular weight compounds. For example, when a TCD and a 10' x 1/8" stainless steel column packed with Carbopack B/3% SP-1500 are used, the water peak interferes with the methanol peak (Figure E1). This interference can be minimized (eliminated in some cases) by using an FID. The FID selectively detects the low molecular weight hydrocarbons while responding minimally to water. When an FID and the same 10' stainless steel column are used, methanol can be quantified (Figure E2). Water does not interfere with components eluting after methanol, whether an FID or a TCD is used.

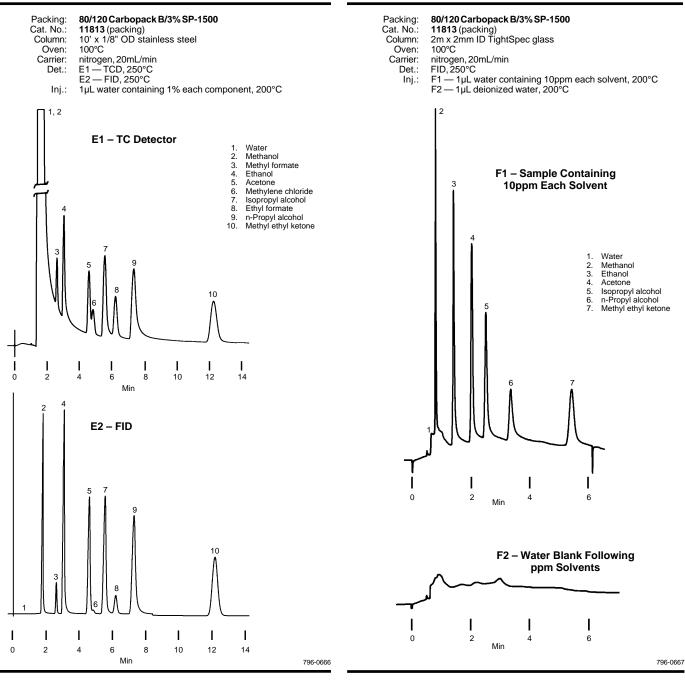
To confirm that Carbopack B/3% SP-1500 columns do not produce memory peaks (**ghosting**), we analyzed aqueous samples

Table 2. Effects of 200 Aqueous Samples on Carbopack B/3% SP-1500 Columns

	Mean for Initial 4 injections	Mean for Last 4 Injections	Ratio of Change (Final:Initial)
Glass Column (2m x 2mm ID)			
Column Efficiency (plates/meter)	2173 ± 109	2209 ± 66	1.02
Column Polarity (Relative retention time of MEK:isopropanol)	1.60 ± 0.00	1.61 ± 0.00	1.01
Total Analysis Time (minutes)	5.47 ± 0.01	5.49 ± 0.01	1.00
Stainless Steel Column (10' x 1/8" OD)			
Column Efficiency (plates/meter)	2043 ± 102	2136 ± 192	1.05
Column Polarity (relative retention time of MEK:isopropanol)	1.67 ± 0.00	1.65 ± 0.00	0.99
Total Analysis Time (minutes)	12.87 ± 0.01	12.97 ± 0.4	1.01

Figure E. Effect of the Detector on Analyses of Solvents in Aqueous Samples

Figure F. No Ghosting with ppm Concentrations of Solvents



containing high (1%) and low (10ppm) concentrations of solvents, following these with injections of pure water. Under conditions appropriate to each analysis, no memory peaks were observed during the analyses of pure water that followed either the 1% or 10ppm (Figure F) levels of solvents. A column used to analyze percent levels of compounds subsequently can be used to analyze ppm levels of compounds. However, much greater detection sensitivity is required. Consequently, the analyst should observe the precautions explained in the next section.

Precautions for Analyzing Trace Levels of Organic Compounds

Certain precautions must be observed prior to and during analyses of sample components at trace levels. Particular care should be taken with column conditioning, use of glass wool plugs, sample size, sources of sample contamination, and detector sensitivity.

Proper **column conditioning** ensures a stable baseline at high detector sensitivities needed in trace analyses. A new column should be programmed from ambient temperature to the upper limit of the stationary phase to bleed off volatile components of the phase. Follow the conditioning instructions that accompany Carbopack B/3% SP-1500 columns and packing.

After this initial conditioning, inject a series of pure water samples onto the column to remove any remaining contaminants and to reduce the size of the water "peak." Figure G demonstrates the effect of water conditioning. During the first injection onto a new Carbopack B 3%/SP-1500 column, water interferes with the methanol peak (Figure G1). By the fifteenth injection this interference decreases significantly, allowing better quantitation of methanol (Figure G2). To immediately obtain quality chromatograms from a new column, condition the column with water (under the conditions you intend to use) until the water peak is acceptable. If the column is used infrequently, repeat this water conditioning accordingly.

For many analyses, silanized **glass wool plugs** are used to retain the packing in the column. However, our studies have shown that silanized glass wool, especially in the column inlet, may adsorb small amounts of sample components. Such adsorption could be significant in trace analyses. In these analyses, therefore, we recommend omitting glass wool from column inlets. When changing the septum or column, carefully depressurize the system to avoid shifting the packing or blowing it out of the column.

Analysts often attempt to compensate for low concentrations of particular sample components by injecting a **large volume of sample**. The column and detector may be able to accept large samples, but often such samples exceed the volume of the injection port. An excessively large sample produces a sudden pressure increase in the injection port, increasing column flow and causing the flow controller to shut down. As the increased pressure dissipates, the flow controller slowly regains control of the column flow. Retention times, however, have decreased because of the slow response. In addition, gases may have been forced back into the carrier gas feed line, dissipating the sample and possibly causing peaks to tail. Eventually the correct flow is reestablished, but retention times and other analytical parameters for the large sample are not reliable.

To illustrate how sample volume affects retention times, we injected 0-10 μ L of distilled water with 1 μ L of an aqueous solution of solvents (0.8% solvents, vol/vol) into a glass column and a

Figure G. Effect of Water Priming on the Water "Peak" from a Carbopack B/3% SP-1500 Column

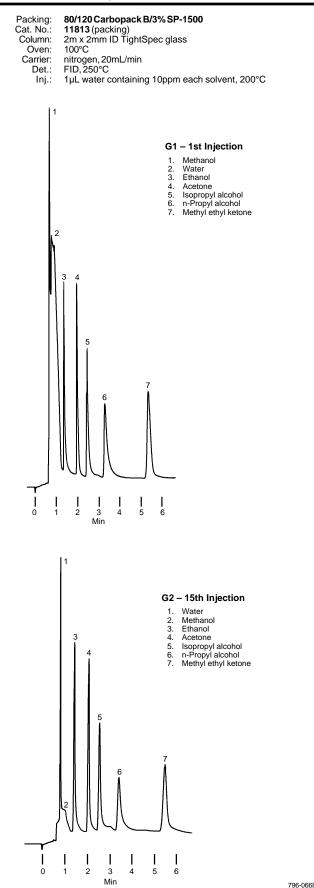


Table 3.Effect of Sample Size on Retention Timesfor Solvents in Water

Sample: Distilled	Abso Retentio	t _R (pentane)/			
Water Volume (µL)	n-Propanol Pentane		t _R (n-propanol)		
Glass Column (2m x 2mm ID)					
1:0 (before)	6.4	12.5	2.1		
1:1	6.5	12.3	2.2		
1:2	6.7	12.3	2.1		
1:4	6.8	12.1	1.8		
1:8	6.8	11.9	1.8		
1:10	7.0	11.5	1.6		
1:0 (after)	6.3	12.5	2.1		
Stainless Steel Colum	Stainless Steel Column (10' x 1/8" OD)				
1:0 (before)	7.0	13.4	1.9		
1:1	7.0	13.2	1.9		
1:2	7.0	13.4	1.9		
1:4	7.0	13.2	1.9		
1:8	7.1	12.9	1.8		
1:10	7.25	13.1	1.8		
1:0 (after)	7.0	13.5	1.9		

stainless steel column, each containing Carbopack B/3% SP-1500. We monitored the retention times for pentane and n-propanol. As progressively more distilled water was injected with the solvent solution, both absolute and relative retention times began shifting (Table 3). The absolute retention data for the glass column shows that the fast eluting n-propanol peak was first to indicate injection port overloading. When a 1:4 mixture was injected, retention times for both compounds were affected. For the stainless steel column, the effect appeared at larger sample volumes. From this data, we concluded that the maximum sample volume for Carbopack B/3% SP-1500 columns should be 2 μ L (containing 0.9ng to 65 μ g of each component) for 2m glass columns or 4 μ L for 10-ft. stainless steel columns.

Contaminants may affect the quantification of sample components. Contamination can occur at several points in the analysis: in glassware used to prepare standards or samples, in the glass injection liner (used in some instruments), or in the syringe used to inject the sample. Good laboratory technique will minimize the possibility of contamination at these sources. All glass surfaces coming in contact with your standards and samples should be as clean as possible. We recommend heating the glassware to remove traces of organics. The GC also should be properly prepared for trace analyses. If your instrument has an injection port liner, we recommend installing a new liner. Most liners are not deactivated by the manufacturer, so be sure to silanize the new liner prior to use. An old liner can be cleaned and baked to remove organics, but still may not provide analyses equal to those obtained with a new liner. Also use a new syringe and reserve it for trace work only, cleaning it carefully between injections. We recommend rinsing the syringe with high purity deionized water, followed by heating and vacuum cleaning in a Hamilton[®] syringe cleaner.

The proper combination of flow rates for carrier gas, hydrogen, and air are necessary to obtain the high **FID sensitivity** needed to analyze samples for trace components. The specific procedure for fine tuning the FID varies among instrument manufacturers, but in general a good starting mixture is approximately 1:1:10 for carrier gas:hydrogen:air. Good initial ranges are 20-30mL/min for carrier gas and hydrogen, and 200-300mL/min for air.

Analysts can use 80/120 Carbopack B/3% SP-1500 columns to monitor many organic compounds as trace components in aqueous solutions. The packing is inert, enabling the user to detect

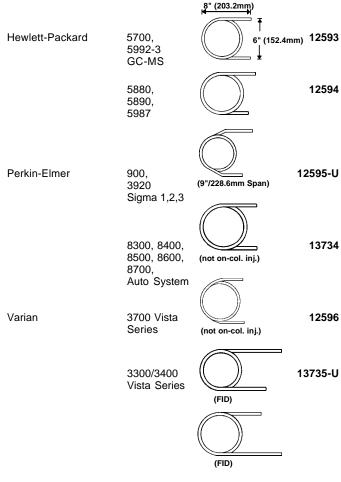
compounds at the maximum sensitivity of an FID. Large amounts of water do not affect packing polarity and stability, and the columns appear to be long lasting. We recommend this packing for the trace work described in this bulletin.

Ordering Information:

Description	Cat. No.
Packing	
80/120 Carbopack B/3% SP-1500, 15g	11813-U

Stainless Steel Packed Columns, 10' x 1/8"

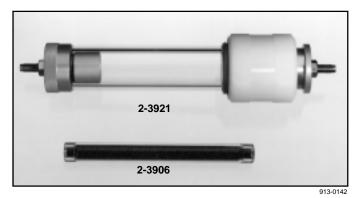
Chromatograph	Model/ Description	Sketch	Cat. No.
General Configuration	` n*		12592



713-0427, 0428, 0429, 0430, 0392, 0431, 0418

*You can carefully bend the column to fit most chromatographs.

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OMI-4 Purifier (tube only)	23909
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*Will not fit OMI-2 tube holder. Use with OMI-1 installation kits.

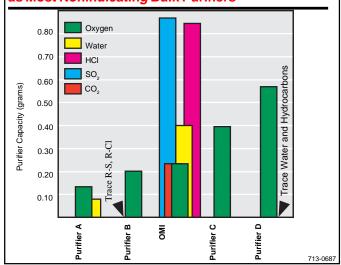
Note: First time users must order both OMI-2 or OMI-4 purifier tube and corresponding holder.

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Water	Yes	No	No
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Carbon dioxide	Yes	No	No
Alcohols/Phenols	Yes	No	No
Sulfur-containing compounds (R-S)	Yes	No	No ***
Halogen-containing compounds (R-CI)) Yes	No	No ***

**If incoming oxygen level does not exceed 10ppm.

*** Corrosive compounds may poison some of these devices.

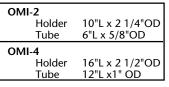


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Dimensions of OMI Purifiers



For more information about gas purification, request Bulletin 848 (Carrier Gas Purification) and publications 411015 (Nanochem Ensures Gas Purity) and 411016 (Stretch Your Carrier Gas Dollars). OMI purifiers contain Nanochem resin, licensed to Supelco, Inc. for use in chromatographic applications. Nanochem is a registered trademark of Matheson Gas Products.

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The stainless steel converter tube is 10" x 1/2" OD. The split-sided heater is 10" long. The unit's integral mounting bracket allows you to bolt the unit to a bench top or wall. The 90 watt power consumption makes the unit as economical to operate as a light bulb.

Unit has 1-year guarantee; elements guaranteed for 90 days.

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1/4" fittings	22398	
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750cc, 1/4" fittings	24564
750cc, 1/2" fittings	24565
Supelcarb Refill, 300cc	24566

Supelpure[™]-HC Hydrocarbon Traps

Activated charcoal adsorbs hydrocarbons and other contaminants from carrier gases, air, and hydrogen. Operates efficiently for approximately six months when total hydrocarbons in the incoming gas average 10ppm.

Description	Cat. No.	
Supelpure-HC Hydrocarbon Traps		
120cc, 1/8" fittings	22445-U	
120cc, 1/4" fittings	22446	
750cc, 1/4" fittings	24518	
750cc, 1/2" fittings	24519	
Charcoal Refill, 400cc	22451	
Mounting Clip, 120cc trap	23993	
Mounting Clip, 750cc trap	24983	

80/120 Carbopack B/3% SP-1500 was developed in cooperation with Dr. A. DiCorcia of the University of Rome.

Reference

1. Gill, J.M. and C.H. Hartmann, J. Gas Chromatogr., 5: 605 (1967). Reference not available from Supelco.

Trademarks

Carbopack, OMI, SP, Supelcarb, Supelco, Supelpure, TightSpec - Supelco, Inc. Cellosolve - Union Carbide Corp. Freon - E.I. du Pont de Nemours & Co., Inc.

Hall — Tracor Instruments, Austin, Inc.

Hamilton - Hamilton Co.

Swagelok — Crawford Fitting Co.

Teflon - E.I. du Pont de Nemours & Co., Inc.

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